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DEFECT-FREE, EDGE POLISHING OF LITHIUM NIOBATE AND OTHER OPTICA--ETC(U)

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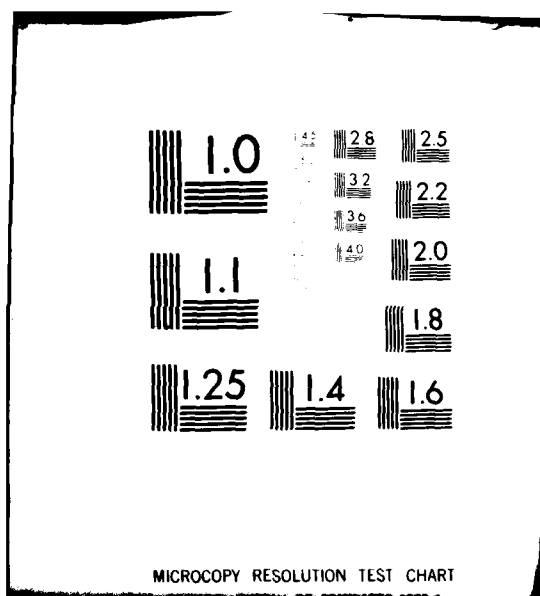
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Technical Report 480

## DEFECT-FREE, EDGE POLISHING OF LITHIUM NIOBATE AND OTHER OPTICAL CRYSTALS

ER Schumacher  
Optics Laboratory  
Code 8115

Final Report: November 1980

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MAR 24 1981  
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ADMINISTRATIVE INFORMATION

The work on this project was accomplished during Fiscal Years '78 and '79 under project 61152N, ZR00001, ZR0210205, 811-ZS1.

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER NOSC Technical Report 480 (TR 480) ✓	2. GOVT ACCESSION NO. AD-A096	3. RECIPIENT'S CATALOG NUMBER 747 (9)
6. TITLE (and Subtitle) DEFECT-FREE EDGE POLISHING OF LITHIUM NIOBATE AND OTHER OPTICAL CRYSTALS		5. TYPE OF REPORT & PERIOD COVERED RTD&E Final Report.
7. AUTHOR(s) E.H. Schumacher		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS Naval Ocean Systems Center San Diego, CA 92152		8. CONTRACT OR GRANT NUMBER(s) 16 17
11. CONTROLLING OFFICE NAME AND ADDRESS 11		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 61152N ZR00001/ZR0210205 811-ZS1
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) 12 16		12. REPORT DATE November 1980
		13. NUMBER OF PAGES 12
		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report)  Approved for public release; distribution unlimited		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)  Optical crystal polishing Edge polishing of Lithium Niobate		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  This report documents a newly developed technique for producing defect-free edge polished lithium niobate sub- strates which overcomes many of the shortcomings associated with previous edge preparation techniques. The technique has been used to make successful, quantitative, repetitive, and sequential optical measurements in guided wave optical devices at the Naval Ocean Systems Center. Step-by-step procedures are provided.		

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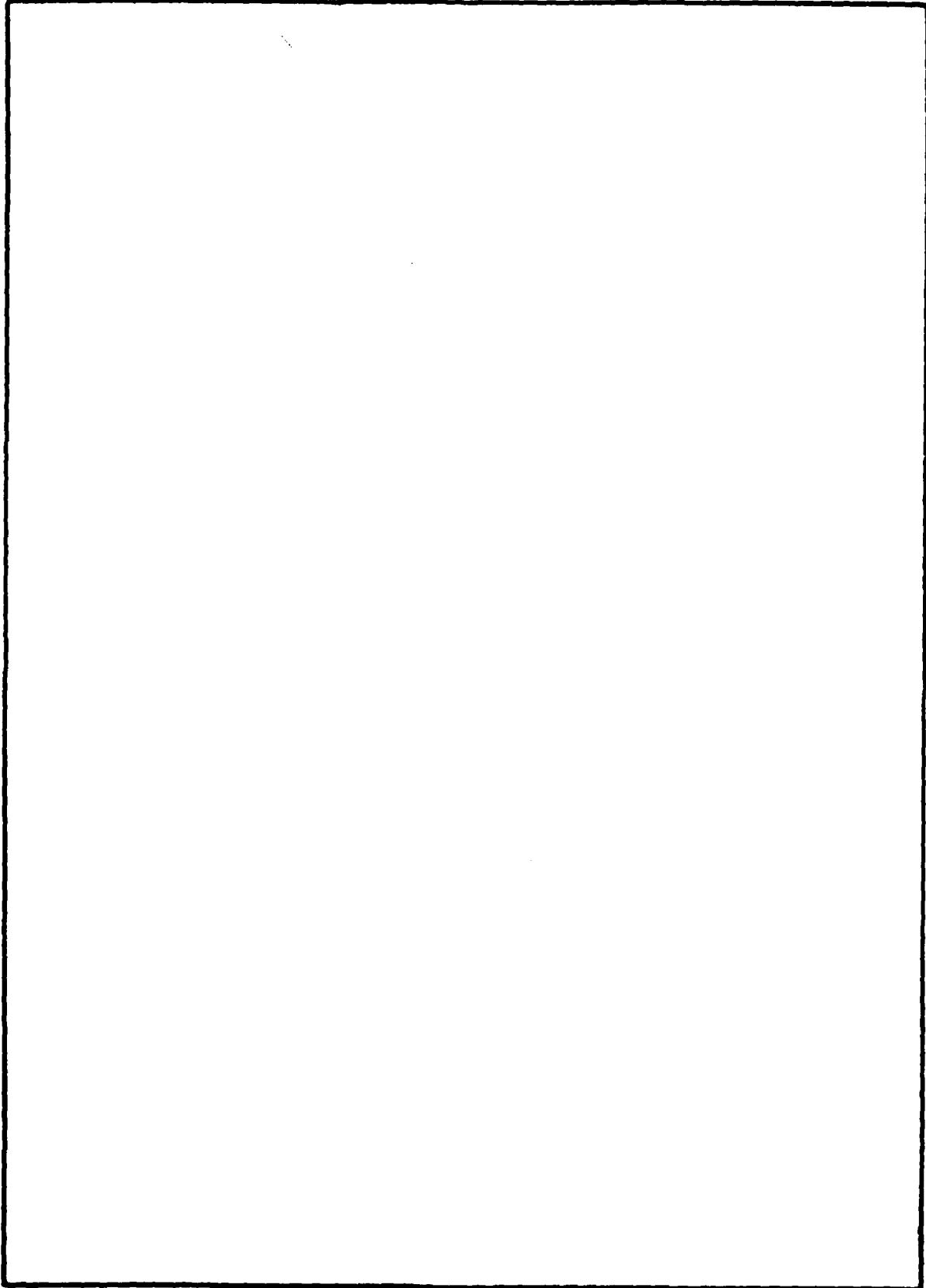
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## INTRODUCTION

The process of cleaving or breaking edges on guided wave optical devices, because of the inherent chipping and directional cleaving characteristics of lithium niobate,<sup>1</sup> causes a large percent of optical energy to be reflected and scattered upon entering and exiting optical waveguides. In the past, these conditions made quantitative optical measurements of guided wave devices difficult and successive or sequential device measurements were almost impossible.

This report documents a newly developed technique for producing defect-free, edge polished lithium niobate substrates which overcomes many of the shortcomings associated with previous edge preparation techniques. The technique has been used to make successful, quantitative, repetitive, and sequential optical measurements in guided wave optical devices at the Naval Ocean Systems Center laboratories. The remainder of this report documents technically the steps involved in edge preparation procedure.

Optical grinding and polishing of  $\text{LiNbO}_3$  wafer edges has been done by Hughes-Malibu, McDonnell-Douglas, Bell Laboratories and others. Discussions regarding various techniques and possible accuracies of edge polishing wafers of lithium niobate took place with H. Taylor, Rockwell International; L. W. Stulz, Bell Laboratories; G. Ramer, Hughes-Malibu; W. Burns, NRL, and G. Burkhart, McDonnell-Douglas. From these discussions, it was determined that the negative characteristics of Y-cut lithium niobate crystals,<sup>1</sup> caused by microscopic hard and soft spots; general strain left over from annealing and local strain induced by frictional heat developed in the polishing process, made the polishing of perfect (no microscopic chips), optically flat edges extremely difficult.

Sample edges were obtained and were microscopically examined (using an 80X, Nomarski Phase Contrast objective at 1000X). Quality of the edges varied as widely as polishing techniques. Edges exhibited serious rollover and chipping was evident. However, it was felt that these edges were an improvement over the cleaving (or breaking) technique and, if some new techniques were incorporated into the process, the edge quality of wafers with practical dimensions could be improved thus permitting uniform measurements, efficient glass fiber to chip coupling and the routine, quantity production of high quality, precision edges. The subsequent five sections document these new techniques.

## PRELIMINARY TOOLING

Because the wafers have to be polished on edge, a special holding or "blocking" piece was fabricated using a round piece of lithium niobate 7 cm OD  $\times$  1.5 cm thick. This piece was diamond blade sawn in half. The two pieces were then cemented together with blocking pitch, flat edge up and a segment 0.5 cm  $\times$  3 cm was diamond generated from each half. The halves were then de-cemented and re-epoxied back together so that a rectangular hole 1 cm  $\times$  3 cm was left in the center. A brass ring 8 cm OD, 7 cm ID, 1.5 cm thick was machined to fit (with a 2 mm lip) over the blocking piece to provide additional strength, safety from chipping and a durable bearing surface for the polishing machine overarm. At this same time a 5 kilogram brass weight was machined 3.5 cm thick, 7.5 cm OD, to fit into the upper part of the ring and an oversized hole was bored to accept the protruding dimensions of the wafers. The weight is needed to produce the friction necessary for rapid diamond polishing.

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<sup>1</sup>K. Nassau, H. J. Levinstein and G. M. Loiacono, J. Phy. Chem. Solids, 27, 983 (1966).

A non-contaminating, fully adjustable overarm was designed and fabricated to accept the blocked wafers and to allow them to rotate freely as they are driven back and forth across the polisher. This overarm was fitted to an existing Strasbaugh random motion optical polishing machine (Model R6Y-DC-1). The blocking piece, driving ring, weight and driving overarm can be seen in figure 1.

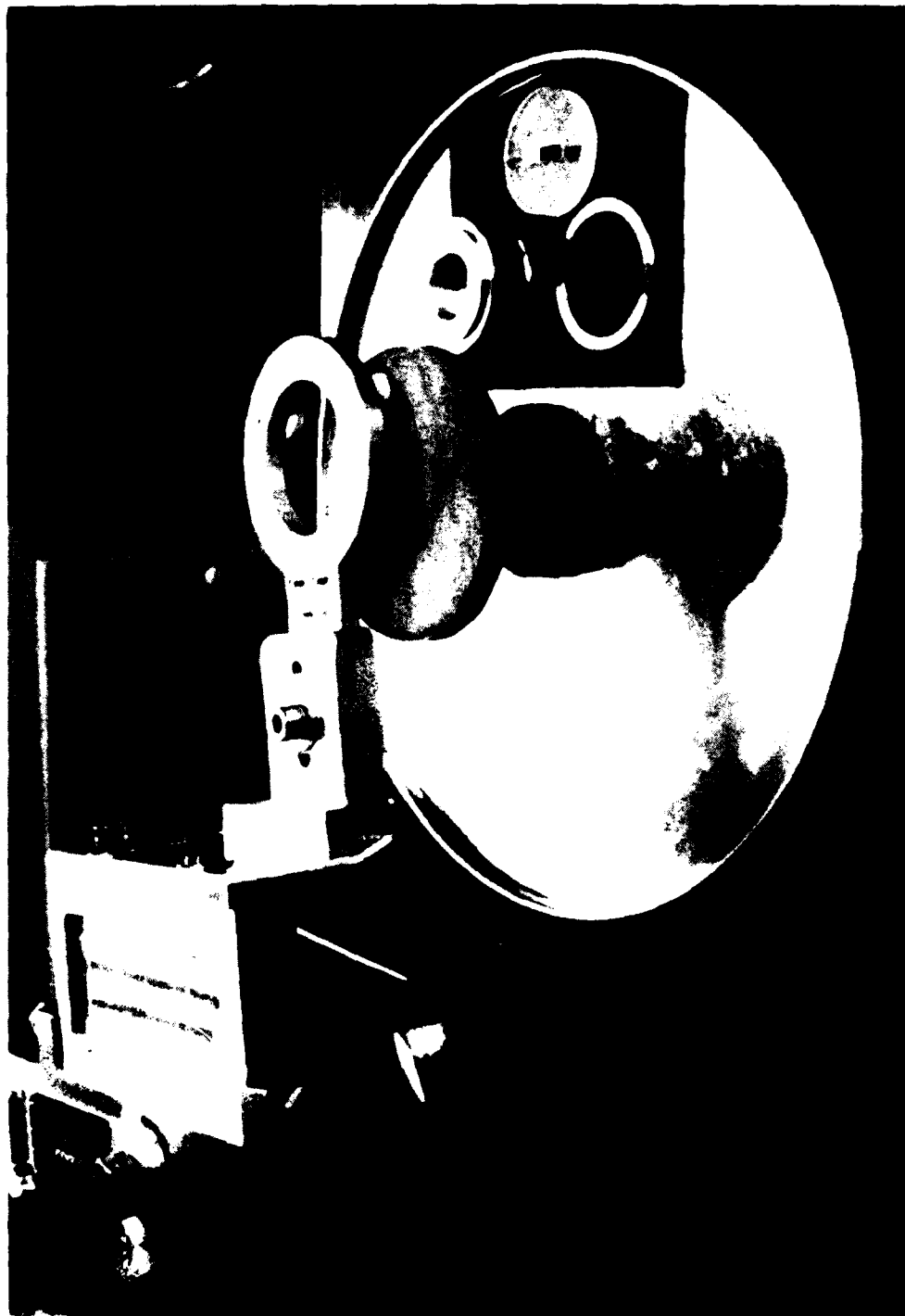


Figure 1.



## BLOCKING WAFERS

The wafers must be blocked or stacked as closely together as possible to prevent large pieces from being fractured from the edges during fine grinding and to prevent the edge surfaces from being rolled over during polishing. Typical spaces between wafers after stacking varied between  $5\ \mu$  and  $22\ \mu$  as measured from four separate stacks of 10 wafers each using a Zeiss standard 20-T reflectance microscope at 800X with a calibrated AO standard eyepiece.

The wafers are normally furnished 5.1 cm OD with a keyed flat edge extending for approximately 3.3 cm, indicating the proper orientation of the wafer axis. (Photolithography is also keyed to this axis.) A blocking jig is constructed so that it makes little contact with the edges and allows the placing of one wafer upon the other without the possibility of rotation or lateral motion.

The wafers, following diffusion, are examined under 150 power and any growth or contamination is removed by mechanical scrubbing followed by a final spin-rinse using filtered isopropanol. The wafers and jig are then placed in an oven and slowly heated to  $120^\circ - 130^\circ\text{C}$ . (A thin piece of aluminum foil is placed between the jig feet and bottom to prevent the cemented assembly from sticking to the jig. After cooling, this foil can easily be peeled from the wafers.) The first wafer is placed in the jig, polished surface up, and 5 to 10 drops of pure, strained, water white rosin are melted (using a Bunsen flame) and allowed to drop on the center of the wafer, one drop directly on the other, forming a single puddle approximately 15 mm in diameter. The next wafer is immediately placed over the first, polished face up, and is allowed to settle without using pressure, until it has wetted its entire surface. The next wafers are then added, using the same technique. When the stack is complete, a small piece of aluminum foil is placed upon the center of the final wafer (or cover piece) and a weight, consisting of a piece of copper rod, 2.5 cm OD and 4 cm long is centered on top of the foil. This assembly is left in the oven at  $125^\circ - 130^\circ\text{C}$  for approximately 2 hours or until all excess rosin has extruded from between the surfaces. Excess rosin is periodically cleaned from the wafers and jig. When this excess no longer flows, the oven is turned off and the assembly left to cool to room temperature. The wafers can then be removed, cleaned and blocked down on a piece of glass, ready for sawing. (A 10 cm OD, 400 grit diamond blade is used.) The blocking pitch consists of 1 part Montan wax, 1 part beeswax and 2 parts rosin. Its melting point is considerably below rosin, preventing the stacked wafers from being disturbed during blocking. After sawing, the block is heated on an insulated hot plate. Once heated, the sawn wafers are quickly removed (in one stack) and the blocked surface only is wiped with a clean cloth. Any chemical cleaning, xylene, acetone, etc., is not permitted beyond this point. (Rosin may be dissolved from between wafers.)

Next, the round lithium niobate blocking piece and its brass driving ring are placed in the oven on top of an aluminum flat covered with aluminum foil and heated to approximately 60°C. A stick of the blocking pitch is held over the rectangular hole and enough pitch is melted to fill the hole approximately 1/4 full. The warmed wafer block is immediately placed edgewise into the hole and the brass driving ring is pitched onto the OD. A small 250 gm weight is placed centrally on the wafers and the entire assembly is removed from the oven and placed upon a cool aluminum surface. Once cooled, the weight is removed, the block is lifted from the flat, the foil peeled and the assembly wiped free of excess pitch. Completed, the assembly should appear as in figure 2 (left). The polishing weight also appears in figure 2 (right) and, when inverted, fits over the blocked assembly.

### GRINDING

It was found that coarse grinding was best accomplished by hand, using a long, elliptical, overhanging stroke on a slowly rotating flat, soft glass (BK-7 or equivalent) tool, 15 cm OD. Grinding began using medium pressure with 14.5  $\mu$  alumina powder and filtered water mixed to a muddy consistency. It continued until the edges (when examined with B&L 0.7-3X StereoZoom microscope) exhibited uniform fracturing. Irregularities from blocking and deep fractures caused by sawing were removed within 30 minutes' time. Grinding pressure then was reduced to zero and another 15 to 20 minutes' grinding took place until improvement could no longer be detected. When finished with 14.5  $\mu$  powder, an average edge, when viewed in a Zeiss 20-T microscope at 625X appeared as in figure 3a. The total time taken was approximately 1 hour and the total material removed was 0.1 mm.

Fine grinding (previous to polishing) was done by hand using 9.5  $\mu$  alumina powder with zero to slight negative pressure. The same glass lap is used along with the same stroke. Care must be taken in this stage to avoid scratching, which occurs when the lap becomes too dry or the grinding mixture too thin. Each wet should be lengthened out as long as possible by the periodic addition of small amounts of filtered water applied with an atomizer while grinding. When the fine grinding is completed (requiring approximately 40 minutes to 1 hour) the total removal should be about 0.05 mm and the edges should appear as in figure 3b and are now ready for polishing.

### POLISHING

Many different polishing surfaces were tried (silk, nylon, teflon, cotton, plastic) in combination with such materials as lacquer, spray plastics, rosin, beeswax, paraffin, pitch and other waxes. Polishing abrasives such as Microgrit aluminas; Cabosil; Linde A, B and C; diamond powders (1/4  $\mu$  through 6  $\mu$  paste and dry); silicon carbide; and Barsite were tried and the following polishing process was found to yield optically flat, defect-free edges in the least possible time.

The rough polishing lap is fabricated using a non-perforated Durlon pad,\* 20 cm OD, cemented to a 20 cm OD, 5 fringe convex (flat) cast iron tool. The polishing surface is charged in a helical pattern with approximately 12 "dots" of 3  $\mu$  diamond compound\*\* from a syringe and worked in with a finger tip. Three or four drops of extender (available from

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\*Available from J. I. Morris Co., Southbridge, Massachusetts 01550, with adhesive back.

\*\*Available from West Coast Diamond Tool Co., Beverly Hills, California, in heavy concentration.



Figure 2.

West Coast Diamond Tool) are spaced equal distances apart and are spread lightly (with finger tip) over the lap. The use of too much extender will cause scratching. The lap is then "broken in" by hand with long straight strokes covering the entire surface while the lap is running at high and low speeds. A piece of scrap  $\text{LiNbO}_3$  3 cm OD  $\times$  0.5 cm thick is used as the "breaking in" tool and the process continued for at least 30 seconds. This process knocks down large particles that cause scratching and it is possible to assess the cutting and polishing action by viewing the "break-in" piece. The lap is now ready for polishing.

The overarm is adjusted for 20 strokes per minute. The spindle speed is set at 130 rpm and the eccentric offset motion is set approximately 2 cm off center. The overarm is

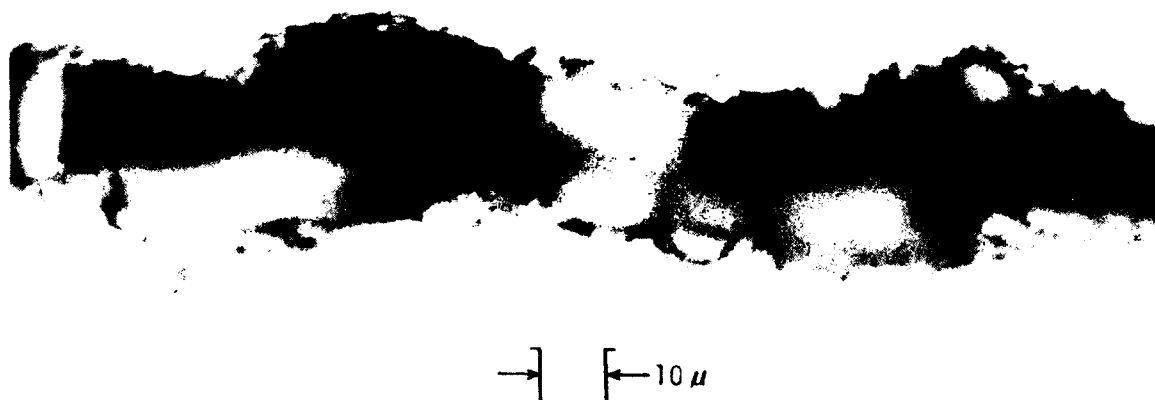


Figure 3a.

set to clear the lap by 0.5 cm. The wafer block is placed gently through the driving hole onto the lap and the brass polishing weight added on top. The machine is turned on and allowed to polish continuously (without attention) for a period of 1 hour.

Following 1 hour's polishing, the block is removed, flushed thoroughly with running tap water and dried gently with clean, absorbent, cotton cloth. The surface should exhibit even, fine sleeks and when viewed at 625X should have the appearance of the edges in figure 3c. Note the sliver shaped piece in the center of figure 3c. This object is a piece of lithium niobate that has finally fallen out from the deeply fractured edge and has imbedded itself in the rosin between the two surfaces. At this point, it is possible to see the difference in roughness between the ground back surface ( $6 \mu$  inch finish) and the polished surface (lower).

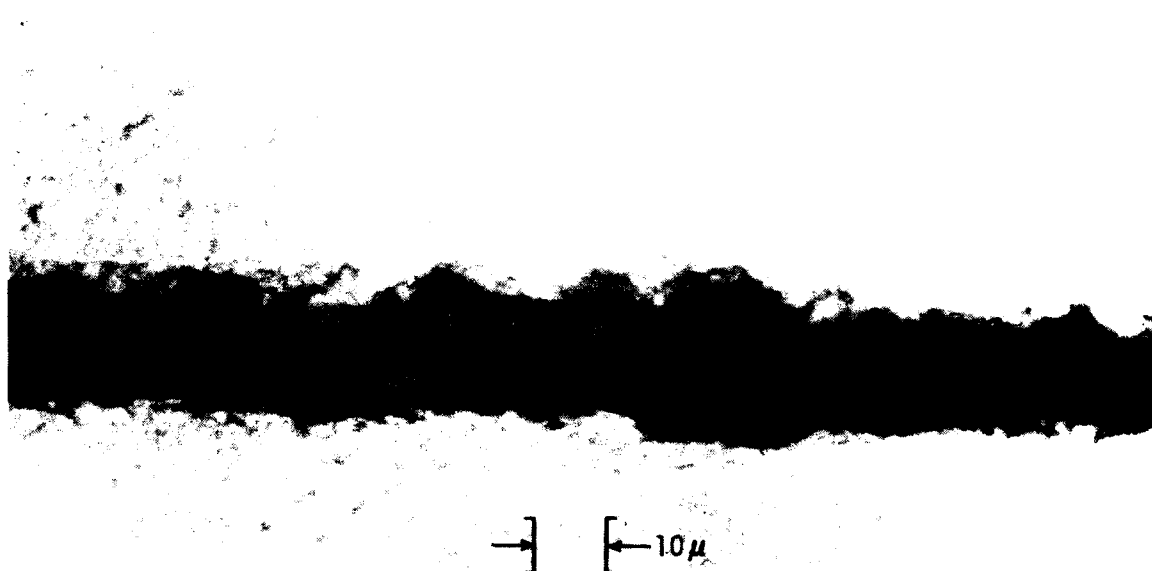


Figure 3b.

The polishing lap is cleaned of the milky appearing "cerf" (or polishing residue) by applying 10 to 15 drops of extender to the lap, equally spaced. This is spread by finger tip around the lap, loosening the cerf. The lap is then wiped clean and dry with a clean, soft absorbent cotton cloth and recharged and "broken in" as previously explained. The "breaking in," polishing, and cleaning process is repeated until no further edge improvement is observed. When the rough ( $3\ \mu$  diamond) polishing is complete, approximately .01 mm material has been removed, requiring approximately 6 hours' polishing time and very little attention. Figures 3c through 3j illustrate the condition of the average wafer edge and include the largest defect. The figure 3 series microphotographs were all taken at 1-hour intervals throughout the polishing process, using a Zeiss 20-T microscope at 625X in conjunction with a Nikon 35 mm F2A camera and a Zeiss 12.5X eyepiece as the camera lens.

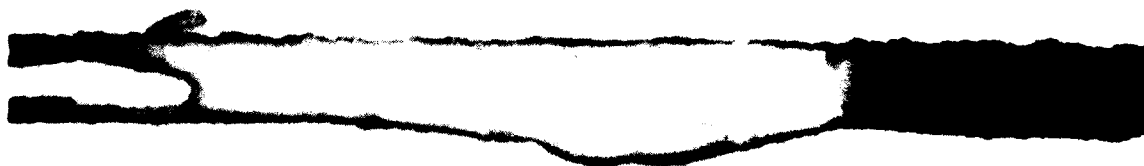
The fine polishing is carried out on three separate laps, one for each grit, all prepared in the following manner: Three 15 cm OD, cast iron, flat tools (3 to 4 fringes convex) are placed in an oven and heated to the point where a stick of pitch (containing 3 parts rosin, 1 part beeswax and 1 part light Montan wax) is easily melted by touching it to the surface of the tool (approximately  $80^{\circ}\text{C}$ ). Once heated, the tools are removed from the oven, placed on a work table and coated with a smooth, thin layer of this pitch. Three 23 cm OD pieces of Nylap\* are then placed over the tools and allowed to soak up the melted pitch. Once this has occurred, the surfaces are squeegeed down, removing all excess pitch and pushing trapped

\*Available from J. I. Morris Co., Southbridge, Massachusetts, without adhesive back.



→ ] [ ← 10  $\mu$

Figure 3c.



→ ] [ ← 10  $\mu$

Figure 3d.

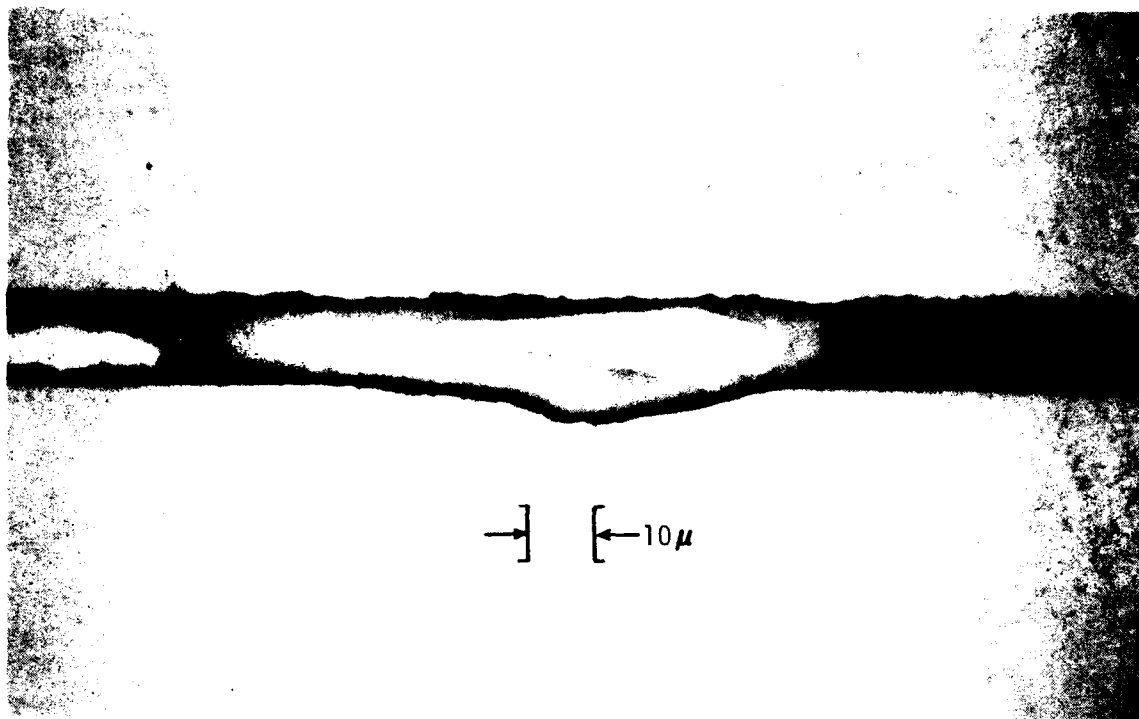


Figure 3e.



Figure 3f.

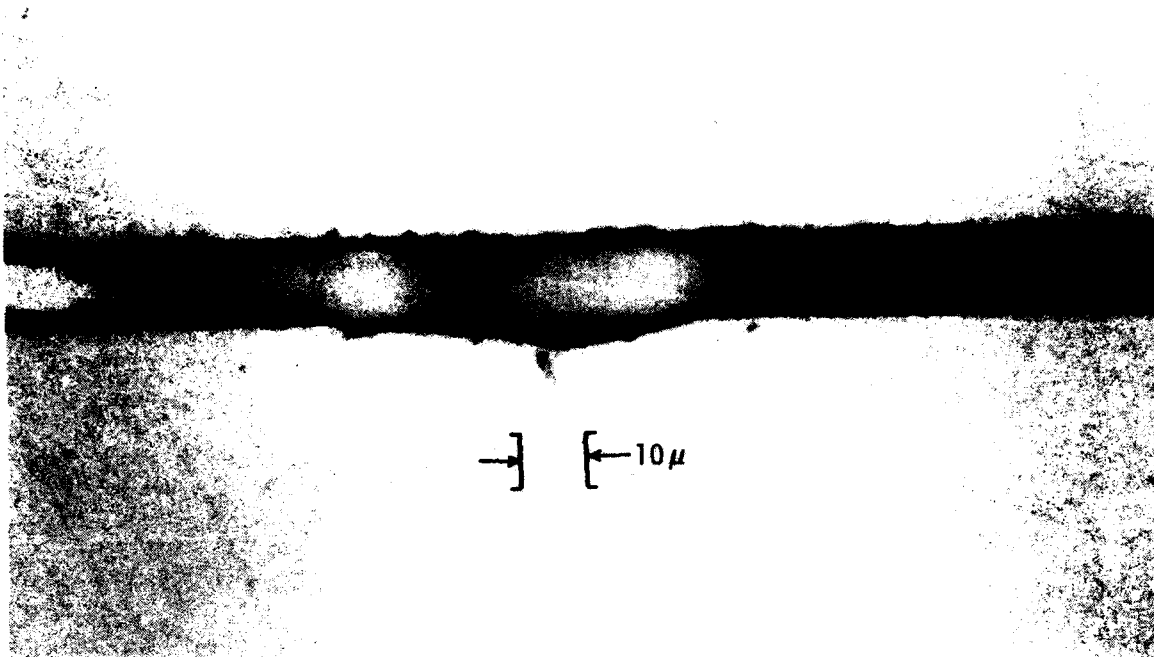


Figure 3g.

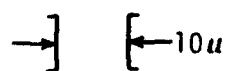


Figure 3h





$\rightarrow ] \quad [ \leftarrow 10 \mu$

Figure 3i.



$\rightarrow ] \quad [ \leftarrow 10 \mu$

Figure 3j.

air bubbles out over the edge. (Enough pitch should be left to fully "set" the Nylap.) The cast iron tools will remain warm for sometime and squeegeeing must continue until the pitch is rigid enough to hold the Nylap down close to the tool surface. These laps are now cooled completely by immersing in filtered tap water. Once cooled, they are closely trimmed at the edge using a new, single edged razor blade taking care not to disturb or fray the fibers around the edge. Extreme care should be taken throughout all these procedures that surfaces be kept clean, smooth and free of all particle contamination. Two of the laps are covered with plastic bags and put aside. The remaining lap is put on the polishing machine ready for charging.

The lap is charged area by area, taking care that too much diamond compound is not used. A dab of  $1\ \mu$  or  $1/2\ \mu$  diamond paste is applied near the edge and, with a clean finger of one hand, thoroughly spread out. Another dab is placed near that area and it too spread out taking care not to overlap areas. (Too many diamonds cannot find "homes" and will migrate over the surface during polishing, causing edge damage by scratching.) The entire surface is prepared in this manner using frugality and conservation. Then, only two or three drops of extender should be applied quickly and lightly to the entire lap surface. Too much extender will dissolve the pitch in the lap. The lap is now ready for the break in procedure previously mentioned in rough polishing (using the same breaking in piece).

Since the lap diameter is now smaller, the length of the overarm stroke is shortened to approximately 7 cm. The speed of the overarm is increased to 50 rpm. The wafer block is inserted through the overarm hold and on to the lap. The brass weight is then placed on top of the wafer block and polishing begun. The spindle speed throughout the entire process is kept at 130 rpm.

Following 1 hour's polishing time, the wafer block is removed, cleaned and inspected in the microscope. The wafer edges should appear as in figure 3i. If they do not, more polishing is required in this stage. The lap should be wiped clean of cerf, using a clean pad of cotton cloth very lightly immersed in xylene. Wiping should be rapid so as not to dissolve too much of the pitch in the lap. The surface once again is charged, broken in and set to polish for another 1-hour period. Once the edge reaches the figure 3i appearance, one may proceed to the next finer grade ( $1/2\ \mu$  or  $1/4\ \mu$  diamonds), using one of the previously prepared laps (new clean surface). Charging, break in and polishing are done in the same manner as previously described, until the wafers' edges reach perfection, as viewed in figure 3j. No visible evidence of edge chipping and little or no evidence of edge turning should be present when viewed through a microscope at 625X. Note: After some use of these laps, frayed edges may begin to appear. To remedy this problem, moisten a clean cotton cloth pad with xylene and rub the pad on the pitch stick, thereby dissolving some of the pitch onto the pad. This process must be continued until the pad has taken on considerable pitch. The pad is then stroked over the polisher surface starting at the center and proceeding over the edge, thereby giving the entire surface a new coat of pitch. (Allow 5 minutes for the xylene to evaporate before using.)

## DE-BLOCKING

Once polished, the wafer block is put into an oven and brought up to a temperature (approximately  $50^{\circ}$ - $60^{\circ}\text{C}$ ) that enables the wafers to be pulled from the block using some force. (The lower temperature prevents the wafers themselves from moving in their stack.) They are then wiped free of blocking pitch (without the use of solvents). The blocking hole is cleaned and the reverse edge of the wafer block is placed back in the hole using previously described techniques.

When the opposite edges have been polished, the wafer block is once again placed in the oven, heated and the wafers pulled from the block. (The use of solvents at and beyond this point is permitted.)

Next, the wafers are placed in a vapor degreaser (see figure 4) so that they are resting on a non-polished edge between two closely spaced holding blocks. (The blocks used were two pieces of copper rod 2.5 cm OD and just exceeded the length of the wafers.) The degreaser is filled with xylene until the wafer tops are approximately 2-3 cm below the surface of the liquid. The degreaser is turned on and set to a temperature that enables the xylene to boil gently (approximately 130°C). The wafers are boiled for a period of 8 hours and left to cool in the xylene overnight.

The next day, the wafers are individually pulled apart, while still immersed, using two small wooden dowels (Q Tip sticks, tapered to a 60° point at one end). Each dowel is placed on a different wafer and some little side force is used to force the wafers apart, one at a time, each time inserting a soft piece of tissue between the newly released wafer and the adjacent wafers. In this manner, the wafers can be de-blocked with practically no surface or edge damage. The wafers can now be gently cleaned of solid residues using favored techniques.

#### SUMMARY

The procedures detailed in this report have been used repeatedly to successfully produce optically flat, microscopically defect-free edges on Y-cut lithium niobate wafers.

These same techniques and materials can be applied in edge polishing other crystalline substrates. The edges produced by these described methods were found to be superior to other edges previously used or examined by NOSC.

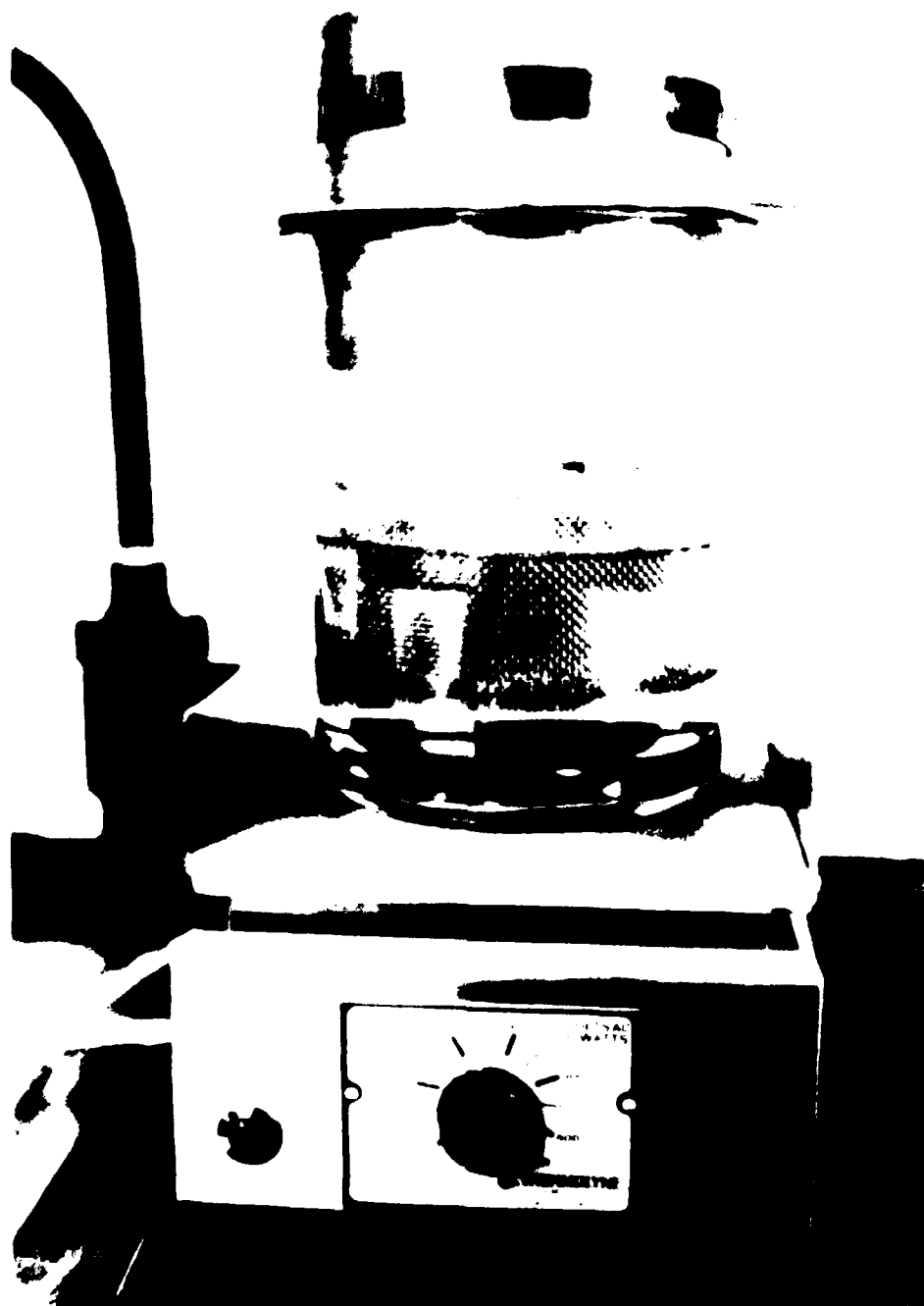


Figure 4.

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